

# The Diamond SQUID

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Diamond is an electrical insulator in its natural form. However, when doped with boron above a critical level ( $\sim 0.25$  at.%) it can be rendered superconducting at low temperatures with high critical fields. Here we present the realization of a micrometer scale superconducting quantum interference device ( $\mu$ -SQUID) made from nanocrystalline boron doped diamond (BDD) films. Our results demonstrate that  $\mu$ -SQUIDS made from superconducting diamond can be operated in magnetic fields as large as 4T independent on the field direction. This is a decisive step towards the detection of quantum motion in a diamond based nanomechanical oscillator.

Micro and nano SQUIDS<sup>1</sup> are extremely sensitive tools for magnetization measurements on the local scale and find applications in various fields of science such as scanning SQUID microscopy<sup>2</sup>. However, the present state of the art limits its utility to magnetic fields well below a tesla. Even though, several attempts to realize micrometer scale superconducting quantum interference devices ( $\mu$ -SQUIDS) form materials with high critical field such as Nb<sub>3</sub>Sn<sup>3</sup> or Nb<sub>3</sub>Ge<sup>4,5</sup> have been realized, the demonstration of a device remaining operational at high magnetic fields has been elusive to date. In this context, diamond, when doped with boron above a critical level ( $\sim 0.25$  at.%) which results in a superconductor with very high critical field<sup>6</sup> is an extremely promising material. In addition, recent advances in diamond thin film growth technology have paved the way toward large scale processing of high quality devices. This recently discovered material not only enables us to make  $\mu$ -SQUIDS capable of operating at fields as high as 4T independent of the field direction, as reported in this paper, but also finds a potential application for ultra-sensitive motion detection of diamond based nanomechanical systems<sup>7</sup>.

The discovery of superconductivity in diamond<sup>6,8–10</sup> has opened the possibility to combine outstanding mechanical properties with superconductivity. Diamond is the archetype of superhard materials with the highest Young's modulus<sup>11,12</sup>. The main interest in such super-hard superconducting materials comes from their possible application to high frequency nanomechanical systems. This high stiffness and the reduced mass of nanomechanical structures made out of diamond enable GHz vibration frequencies comparable with or higher than thermal energies at milliKelvin temperatures<sup>13</sup>. This opens the possibility of studying the quantum regime of such nanomechanical resonators<sup>14–16</sup>.

Advances in nanofabrication technology have made it possible to realise complex devices involving nano-electromechanical components<sup>17,18</sup>. But in most cases

the nano-mechanical devices are compound/hybrid devices consisting of a super-hard component with high Young's modulus generating high frequency resonators, and of a conducting component to reduce the transmission losses of the system. However, this conductive component decreases the mechanical rigidity and thus reduces the overall quality factor of the device. In this context, boron doped diamond turns out to be an excellent candidate for fabricating monolithic superconducting circuits involving nanomechanical systems with very high quality factor. One can then envision the direct integration of such nanomechanical systems into superconducting circuits such as SQUIDS<sup>7</sup> or superconducting resonators<sup>19</sup> for ultra sensitive motion detection.

## Results and Discussions

As a first step in this direction, we fabricated diamond  $\mu$ -SQUIDS patterned from a 300 nm thick superconducting nano-crystalline diamond film. The boron doped diamond films were grown by microwave plasma enhanced chemical vapour deposition (MWPECVD) on seeded silicon 100 wafers with a silica buffer layer of 500 nm<sup>20</sup>. The thickness was monitored in situ with laser interferometry. The growth process has been discussed in detail elsewhere<sup>21</sup>. The diamond thin films were patterned using standard electron beam lithography. A thin nickel mask (65 nm) was deposited for subsequent highly anisotropic oxygen plasma etching<sup>22</sup>. Titanium-platinum-gold was deposited for the contact pads (figure 1a) and the sample was annealed at 750° C to ensure good ohmic properties.

We fabricated several similar devices as depicted in figure 1 with various weak link designs of width of 250 nm, 170 nm and 100 nm. These weak links serve as the Josephson junctions in the superconducting loop<sup>23,24</sup>. Here we mainly discuss data measured on a  $\mu$ -SQUID with an area of  $2.5\mu\text{m} \times 2.5\mu\text{m}$  with two symmetric weak links (100 nm wide if not stated otherwise). Our low field measurements were performed in a <sup>3</sup>He closed cycle refrigerator with a base temperature of 400 mK, whereas

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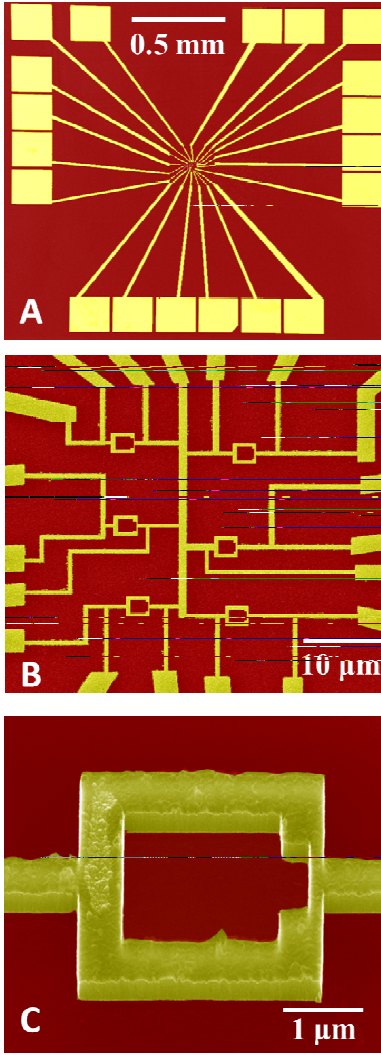


FIG. 1: Scanning electron micrograph of the  $\mu$ -SQUID circuit. a, Image of the complete diamond circuit showing several pads for ohmic contacts. The total chip size is  $2\text{mm} \times 2\text{mm}$ . b, Close-up view of a  $50 \times 50 \mu\text{m}^2$  area in the middle of the sample. The circuit contains six  $\mu$ -SQUIDs with different geometrical characteristics. c, Tilted view of one of the  $\mu$ -SQUIDs. The mean loop area of all the  $\mu$ -SQUIDs is  $2.5 \times 2.5 \mu\text{m}^2$ . The thickness of the diamond film is 300 nm while the arms of the  $\mu$ -SQUIDs are 500 nm wide. Two weak links of 170 nm in width and 250 nm in length can be identified.

the high field measurements have been undertaken in a dilution refrigerator at a temperature of 40 mK. The superconducting critical temperature of the bulk as well as the nanostructured diamond film was about 3K as shown in figure 2a.

The general characteristics of our  $\mu$ -SQUIDS have been summarized in figure 2. The current voltage (I-V) characteristic shows a thermal hysteresis<sup>25,26</sup> with a critical current of almost  $1\mu\text{A}$  and a retrapping current of about  $0.55\mu\text{A}$ . These parameters can be changed by tuning the geometrical aspects of the weak link. To demonstrate

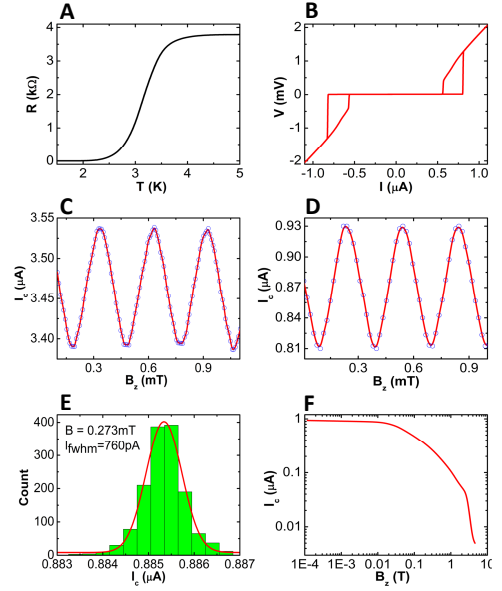


FIG. 2: Characteristic features of the diamond  $\mu$ -SQUID. a, Superconducting transition of our  $\mu$ -SQUID showing a transition temperature around 3K which is close to the  $T_C$  of the diamond thin film. b, Current voltage characteristics of the  $\mu$ -SQUID with 100 nm wide weak link. The I-V curve is hysteretic with a critical current close to  $1\mu\text{A}$  and a retrapping current of about  $0.55\mu\text{A}$ . c, d) Low field oscillation of the critical current as a function of magnetic field for a  $\mu$ -SQUID with a width of a weak link of 170 nm and 100 nm, respectively. The oscillation period in both cases is around 0.31 mT. e, Histogram of the switching current of the  $\mu$ -SQUID with a 100 nm wide weak link. The measurement was done by recording the switching current repetitively at a fixed magnetic field of 0.3 mT. f, Field dependence of the critical current for the same  $\mu$ -SQUID in perpendicular configuration.

the performance of our diamond  $\mu$ -SQUIDS we have measured the critical current oscillation as a function of perpendicular magnetic field. At low magnetic field we swept the current from a value slightly below the retrapping current until the switching of the SQUID was detected and recorded. This was repeated for different magnetic fields. The results for these experiments are shown in figure 2c and d. An oscillation period of approximately 3.1 G was recorded which corresponds to an effective SQUID surface area of  $2.6 \times 2.6 \mu\text{m}^2$ , in agreement with the geometrical dimensions of the  $\mu$ -SQUID. The resulting modulation amplitude was about 15% for 100 nm wide weak links and around 5% for a 250 nm width comparable to what is observed for Al and Nb  $\mu$ -SQUIDS<sup>27</sup>. To probe the sensitivity of our  $\mu$ -SQUID we repeated current switching measurements at a fixed magnetic field. Taking the full width at half maximum and taking into account the sampling frequency we obtained a sensitivity of  $4 \times 10^{-5} \phi_0 / \sqrt{Hz}$ , where  $\phi_0$  is the superconducting flux quantum. This sensitivity is comparable to similarly designed  $\mu$ -SQUIDS from frequently employed superconductors such as niobium or aluminium<sup>28</sup> but a careful

optimization of the SQUID design as well as material can lead to much higher sensitivities<sup>29</sup>. At present the sensitivity is only limited by the electronic measurement set-up or external noise as the histograms are still symmetric and not limited by quantum fluctuations even at 40 mK. The main advantage of the diamond system is that superconductivity persists up to very high magnetic field (in our case more than 4 T). This is shown by the field dependence of the critical current as depicted in figure 2f.

Various types of  $\mu$ -SQUIDs have been reported in the literature, which are extremely sensitive magnetic flux detectors and currently used for scanning SQUID microscopy<sup>1,28</sup>, magnetization measurements in mesoscopic systems<sup>30,31</sup> and in isolated molecules<sup>32</sup>. However, a severe drawback of these detectors is the narrow field range in which they are operable. In particular,  $\mu$ -SQUID devices were operated at fields above 1 T only when the field is applied perfectly in the plane of the SQUID<sup>33</sup>. In addition, in such a parallel field configuration, the thickness of the superconducting layer has to be extremely small (of the order of few nanometers), a feature which drastically reduces the critical current. On the other hand, operating *standard*  $\mu$ -SQUIDs in perpendicular field reduces severely the operational field range well below 1 T. Here we demonstrate that  $\mu$ -SQUIDs made from boron doped diamond do not suffer from such limitations and can be operated in magnetic fields as high as 4 T even when applied in perpendicular configuration. This is more than a six-fold increase on the present state of the art<sup>34</sup>.

In figure 3c-f we have shown the characteristic SQUID oscillations at various fields up to 4 T. The oscillations are not perfectly periodic due to the fact that we had to use the z-coil (perpendicular to SQUID plane) for both, to apply the steady magnetic field (up to 4 T) and also to probe the SQUID oscillations (of the order of 0.3 mT). The field resolution is hence limited by the resolution of the magnet power supply ( $\sim 0.1$  mT). This could be improved by adding an additional feed line next to the  $\mu$ -SQUID to probe the SQUID oscillations independently<sup>7</sup>.

In order to demonstrate the insensitivity of our SQUID to the applied field direction we have also measured SQUID oscillations by applying a constant field with a vector magnet in the x-y plane (parallel to SQUID plane). In this configuration the SQUID oscillations were probed using a small magnetic field of a few milli Teslas in the z-coil. The voltage oscillations for a parallel field of 0.5 and 1 T, the maximal field achievable with our vector coil, are shown in figure 4. Let us also emphasize that these oscillations were obtained for 300 nm thick superconducting boron doped diamond layers. In this case the aspect ratio is very favorable for vortex penetration, and in standard superconductors, current oscillations are usually not observed in  $\mu$ -SQUIDS for layers thicker than a few nanometers. The fact that we are able to observe SQUID oscillations for such geometries may originate from the granularity of the material<sup>35</sup>, which favors vor-

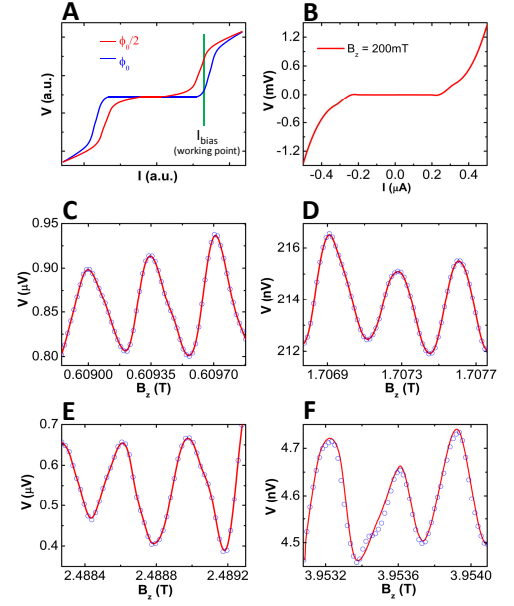


FIG. 3: SQUID characteristics in perpendicular field configuration. a, The schematic of two I-V curves in the non-hysteretic regime of the device for a applied magnetic flux of  $\phi_0$  and  $\phi_0/2$ . By current biasing the device at the green line we can record the oscillation in the output voltage as the applied magnetic field is swept. b, I-V characteristic when the SQUID is in the non-hysteretic regime. c-f, SQUID oscillations at various field ranges (C $\sim$ 0.6T, D $\sim$ 1.7T, E $\sim$ 2.5T, F $\sim$ 4T). The average oscillation period is 3.2G, consistent to what is observed at low magnetic field. The contrasts were not optimized in these experiments and hence do not follow the field dependence of the critical current.

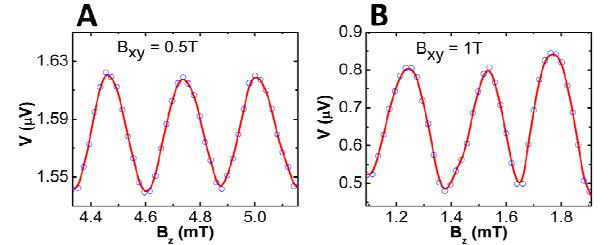


FIG. 4: SQUID characteristics in parallel field configuration. a and b, The voltage oscillations of the SQUID when a constant magnetic field is applied parallel to the SQUID plane and perpendicular to both the weak links. To detect the oscillation a small probing field is applied perpendicular to the SQUID plane.

tex pinning.

Let us mention that the resulting SQUID oscillations at high magnetic field have not been optimized at each field and for this reason the oscillation amplitude does not follow the field strength. Here we simply demonstrate the proof of principle that a diamond SQUID can be operated at high magnetic field and this independent on the field direction. In order to optimize the sensitivity of the diamond SQUID it would be extremely interesting

to employ single crystal diamond where higher critical currents<sup>36</sup> as well as higher critical fields ( $\sim 10\text{T}$ )<sup>6</sup> can be achieved.

SQUID oscillations surviving at high magnetic fields, independent of field orientation, open the possibility to exploit these exceptional properties for HF magnetic surface probe techniques<sup>2</sup>. Combining these exceptional superconducting and outstanding mechanical properties makes this monolithic system a highly promising tool, in particular for the detection of quantum motion in a diamond based nanomechanical oscillator when inserted within one arm of a diamond SQUID<sup>7</sup>.

## Methods

### Film Deposition

Prior to deposition, the wafers were cleaned by standard RCA SC1 solution and rinsed with deionized water in an ultrasonic bath. Immediately after rinsing the wafers were immersed in a colloid of mono-disperse diamond nano-particles known to have a mean size of 6 nm, and agitated by ultrasound for 30 min. Following this seeding process, the wafers were rinsed again with deionized water, blown dry in nitrogen and immediately placed inside the growth chamber which was pumped down to a vacuum lower than  $10^{-6}$  mbar. The chamber was then purged with hydrogen gas and a plasma ignited with 4% methane and 6500 ppm of tri-methylboron diluted by hydrogen (better than 99.9999999% pure). The pressure was ramped to 50 mbar and the temperature was  $700^\circ\text{C}$  as measured by optical pyrometry.

### Electronic Measurements

The SQUID measurements have been performed by current biasing the squid loop via a thermostable 1 M $\Omega$  resistor using a 16 bit NI-USB-6229 DAC. At low magnetic field, the I-V characteristic is hysteretic as seen in figure 2b and the SQUID oscillation is measured by recording the critical current as a function of magnetic

field. The critical current is determined when a voltage drop is generated across the SQUID due to its transition from the superconducting to the normal state. The voltage is amplified with a NF-LI75a low-noise voltage amplifier and recorded via a Keithley 2000 Multimeter. The SQUID oscillations shown in figure 2 are obtained with a single I-V curve at a fixed magnetic field. No averaging has been done.

Applying a large magnetic field ( $B > 500\text{ mT}$ ) to a superconductor reduces the critical current. When the reduction of the critical current is below the value of the re-trapping current, the voltage-current characteristics become non-hysteretic as shown in figure 3b. In this case, an ac measurement technique can be used to measure the SQUID oscillations. The SQUID is biased with a square wave signal by mixing a sinusoidal voltage (amplitude of 1 V and a frequency of 11.7 Hz) with the dc voltage of the NI-DAC. The voltage generated across the SQUID is amplified with a home-made ultra-low noise voltage amplifier ( $0.5\text{nV}/\sqrt{\text{Hz}}$ ) and measured with a Lock-In amplifier (Signal recovery 7265). An I-V curve measured with this technique is displayed in figure 3b. By adjusting the DC voltage of the DAC, the bias current  $I_B$  can then be set at the working point of the SQUID (see figure 3A). Sweeping the magnetic field at fixed bias current results in voltage oscillations as shown in figures 3c-f and 4a,b.

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